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UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No.	1999-385-1
First Inventor	Frederick F. Lange
Title	* see first page of specification
Express Mail Label No.	EJ502972532US

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, DC 20231

1. [x] Fee Transmittal Form (e.g., PTO/SB/17)
   (Submit an original and a duplicate for fee processing)
2. [x] Applicant claims small entity status.
   See 37 CFR 1.27.
3. [x] Specification   [*Total Pages* [17]]
   (preferred arrangement set forth below)
   - Descriptive title of the invention
   - Cross Reference to Related Applications
   - Statement Regarding Fed sponsored R & D
   - Reference to sequence listing, a table,
     or a computer program listing appendix
   - Background of the Invention
   - Brief Summary of the Invention
   - Brief Description of the Drawings (if filed)
   - Detailed Description
   - Claim(s)
   - Abstract of the Disclosure
4. [x] Drawing(s) (35 U.S.C. 113)   [*Total Sheets* [17]]
5. Oath or Declaration   [*Total Pages* [3]]
   a. [x] Newly executed (original or copy)
   b. [ ] Copy from a prior application (37 CFR 1.63 (d))
         (for continuation/divisional with Box 17 completed)
      i. [ ] **DELETION OF INVENTOR(S)**
         Signed statement attached deleting inventor(s)
         named in the prior application, see 37 CFR
         1.63(d)(2) and 1.33(b).
6. [ ] Application Data Sheet. See 37 CFR 1.76

7. [ ] CD-ROM or CD-R in duplicate, large table or
       Computer Program (*Appendix*)
8. Nucleotide and/or Amino Acid Sequence Submission
   (*if applicable, all necessary*)
   a. [ ] Computer Readable Form (CRF)
   b. Specification Sequence Listing on:
      i. [ ] CD-ROM or CD-R (2 copies); or
      ii. [ ] paper
   c. [ ] Statements verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

9. [x] Assignment Papers (cover sheet & document(s))
10. [ ] 37 CFR 3.73(b) Statement   [x] Power of
        (when there is an assignee)   Attorney
11. [ ] English Translation Document (*if applicable*)
12. [x] Information Disclosure   [x] Copies of IDS
        Statement (IDS)/PTO-1449   Citations
13. [ ] Preliminary Amendment
14. [x] Return Receipt Postcard (MPEP 503)
        (Should be specifically itemized)
15. [ ] Certified Copy of Priority Document(s)
        (if foreign priority is claimed)
16. [ ] Other: ....
         ....

17. If a CONTINUING APPLICATION, *check appropriate box, and supply the requisite information below and in a preliminary amendment, or in an Application Data Sheet under 37 CFR 1.76:*

[ ] Continuation   [ ] Divisional   [ ] Continuation-in-part (CIP)   of prior application No :_____ /_____

Prior application information: Examiner _____   Group / Art Unit: _____

For CONTINUATION OR DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or declaration is supplied under Box 5b, is considered a part of the disclosure of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.

18. CORRESPONDENCE ADDRESS

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Signature	*[signature]*	Date	September 29, 2000

JC930 U.S. PTO 09/29/00

JC825 U.S. PTO 09/670918 09/29/00

FEE TRANSMITTAL
for FY 2000

Patent fees are subject to annual revision.

Complete if Known	
Application Number	
Filing Date	September 29, 2000
First Named Inventor	Frederick F. Lange
Examiner Name	
Group Art Unit	
Attorney Docket No.	1999-385-1

TOTAL AMOUNT OF PAYMENT ($) 902

METHOD OF PAYMENT (check one)

1. [X] The Commissioner is hereby authorized to charge indicated fees and credit any overpayments to:

Deposit Account Number: 21-0649

Deposit Account Name: Regents of U. of California

[X] Charge Any Additional Fee Required Under 37 CFR 1.16 and 1.17

[X] Applicant claims small entity status See 37 CFR 1.27

2. [ ] Payment Enclosed:
[ ] Check [ ] Credit card [ ] Money Order [ ] Other

FEE CALCULATION

1. BASIC FILING FEE

Large Entity		Small Entity		Fee Description	Fee Paid
Fee Code	Fee ($)	Fee Code	Fee ($)		
101	690	201	345	Utility filing fee	345
106	310	206	155	Design filing fee	
107	480	207	240	Plant filing fee	
108	690	208	345	Reissue filing fee	
114	150	214	75	Provisional filing fee	

SUBTOTAL (1) ($) 345

2. EXTRA CLAIM FEES

	Extra Claims		Fee from below	Fee Paid
Total Claims	63	-20** = 43	x 9	= 387
Independent Claims	1	- 3** = 0	x 0	= 0
Multiple Dependent			130	= 130

**or number previously paid, if greater; For Reissues, see below

Large Entity		Small Entity		Fee Description
Fee Code	Fee ($)	Fee Code	Fee ($)	
103	18	203	9	Claims in excess of 20
102	78	202	39	Independent claims in excess of 3
104	260	204	130	Multiple dependent claim, if not paid
109	78	209	39	** Reissue independent claims over original patent
110	18	210	9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) ($) 517

FEE CALCULATION (continued)

3. ADDITIONAL FEES

Large Entity		Small Entity		Fee Description	Fee Paid
Fee Code	Fee ($)	Fee Code	Fee ($)		
105	130	205	65	Surcharge - late filing fee or oath	
127	50	227	25	Surcharge - late provisional filing fee or cover sheet	
139	130	139	130	Non-English specification	
147	2,520	147	2,520	For filing a request for *ex parte* reexamination	
112	920*	112	920*	Requesting publication of SIR prior to Examiner action	
113	1,840*	113	1,840*	Requesting publication of SIR after Examiner action	
115	110	215	55	Extension for reply within first month	
116	380	216	190	Extension for reply within second month	
117	870	217	435	Extension for reply within third month	
118	1,360	218	680	Extension for reply within fourth month	
128	1,850	228	925	Extension for reply within fifth month	
119	300	219	150	Notice of Appeal	
120	300	220	150	Filing a brief in support of an appeal	
121	260	221	130	Request for oral hearing	
138	1,510	138	1,510	Petition to institute a public use proceeding	
140	110	240	55	Petition to revive - unavoidable	
141	1,210	241	605	Petition to revive - unintentional	
142	1,210	242	605	Utility issue fee (or reissue)	
143	430	243	215	Design issue fee	
144	580	244	290	Plant issue fee	
122	130	122	130	Petitions to the Commissioner	
123	50	123	50	Petitions related to provisional applications	
126	240	126	240	Submission of Information Disclosure Stmt	
581	40	581	40	Recording each patent assignment per property (times number of properties)	40
146	690	246	345	Filing a submission after final rejection (37 CFR § 1.129(a))	
149	690	249	345	For each additional invention to be examined (37 CFR § 1.129(b))	
179	690	279	345	Request for Continued Examination (RCE)	
169	900	169	900	Request for expedited examination of a design application	

Other fee (specify) _____

* Reduced by Basic Filing Fee Paid

SUBTOTAL (3) ($) 40

SUBMITTED BY

Name (Print/Type)	Tim W. Wan	Registration No. (Attorney/Agent)	45,421	Telephone	(510) 587-6071
Signature	*Tim. Wan*			Date	September 29, 2000

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STATEMENT CLAIMING SMALL ENTITY STATUS (37 CFR 1.9(f) & 1.27(d))--NONPROFIT ORGANIZATION	Docket Number (Optional) 1999-385-1

Applicant, Patentee, or Identifier: **Frederick F. Lange**

Application or Patent No.: **Not Yet Assigned**

Filed or Issued: **Herewith**

Title: **METHOD FOR IMPROVING THE RELIABILITY OF BRITTLE MATERIALS THROUGH THE CREATION OF A THRESHOLD STRENGTH**

I hereby state that I am an official empowered to act on behalf of the nonprofit organization identified below:

NAME OF NONPROFIT ORGANIZATION **The Regents of the University of California**

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☒ the specification filed herewith with title as listed above.
☐ the application identified above.
☐ the patent identified above.

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NAME OF PERSON SIGNING **Tim W. Wan**

TITLE IN ORGANIZATION OF PERSON SIGNING **Licensing Associate, Office of Technology Transfer**

ADDRESS OF PERSON SIGNING **1111 Franklin Street, 5th Floor, Oakland, CA 94607-5200**

SIGNATURE _(signature)_ DATE **September 29, 2000**

METHOD FOR IMPROVING THE RELIABILITY
OF BRITTLE MATERIALS THROUGH THE CREATION OF
A THRESHOLD STRENGTH

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

BACKGROUND OF THE INVENTION

Field of Invention

This discovery concerns the improvement of the reliability and damage tolerance of brittle materials through the use of novel crack arresting architectures composed of compressive layers specifically placed throughout the body that prevent failure from occurring until a specific, predetermined threshold strength is reached.

Description of Related Art

The strength of most common brittle materials is not deterministic, i.e. single-valued, due to the presence of an unknown distribution of strength-limiting flaws inadvertently introduced during processing and surface machining [1,2]. As a result, the strength of brittle materials must generally be described by a statistical distribution of strengths with associated probabilities of failure at each of those strengths. Failure from these types of flaws is generally not an issue in ductile materials because they exhibit plastic deformation that desensitizes the relation between small flaws and strength. Plastic deformation also absorbs work from the loading system to increase the material's resistance to the extension of large cracks. However, the lack of plastic deformation in brittle materials causes their strength to be inversely dependent on the size of very small cracks, which generally cannot be detected except by failure itself.

Consequently, design with brittle materials generally becomes a practice of defining acceptable levels of reliability. Designers must not only make accommodations for probabilistic definitions of the strength and the finite probability of failure at any applied stress, but they must also be further concerned with the fact that, once in service, seemingly insignificant and sometimes undetectable damage could be incurred that would drastically reduce the load carrying ability of the material. This lack of reliability is one of the major reasons why brittle materials have not been more widely used, despite the potential they offer for substantial performance enhancements in a wide variety of applications.

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One method for improving the reliability of components made from brittle materials has been through the use of proof testing. The proof test is designed to emulate the thermomechanical stresses experienced by the component in severe service and defines a threshold stress below which components are eliminated by failure prior to service. However, given its destructive nature, proof testing is generally only used when performance needs outweigh consumer price sensitivity. In ceramics, another approach to ensuring reliability is by eliminating heterogeneities that give rise to flaws, such as inclusions and agglomerates, from the ceramic powder. One method to remove heterogeneities greater than a given size is to disperse the powder in a liquid and pass the slurry through a filter [1]. If heterogeneities are not reintroduced in subsequent processing steps, and surface cracks introduced during machining are not a critical issue, filtration determines a threshold strength by defining the largest flaw that can be present in the powder and thus, within the finished ceramic component [3]. However, neither of these techniques mitigates the detrimental effect of service-related damage.

Recently, another method for improving reliability through the use of residual, compressive stresses that have their maxima located some specific distance beneath the surface of the material was proposed by Green et al [4]. The authors suggested that the unique compressive stress profiles they developed would arrest surface cracks and lead to higher failure stresses and improved reliability through reduced strength variability. However, compressive stresses, either at or just beneath the surface, will not effectively hinder internal cracks and flaws, nor can they produce a threshold strength; thus high reliability is still not ensured. As shown below, a threshold strength can only arise when compressive layers are placed on the surface *and* throughout the body to interact with both surface cracks and internal cracks and flaws.

SUMMARY OF THE INVENTION

The present invention provides a new method for fabricating reliable, damage-tolerant brittle materials. By incorporating layers of one material under residual compression on the surface and throughout the bulk of one or more other materials, a composite is formed in which the propagation of otherwise catastrophic cracking is arrested. The residual compression within these layers acts to reduce the stress intensity of the cracks, thereby causing them to arrest until further loading is provided. This highly desirable stable, subcritical crack growth mode persists with increased loading until the applied stress is large enough to drive the crack completely through compressive region, after which failure occurs.

The exact level of stress needed to cause failure is dictated by the architectural design of the compressive layers such that the material can be designed to have any minimum strength desired, within the limits of the materials system used. This results in a truncation of the strength distribution, such that there is virtually zero probability of failure below this minimum value, i.e. the material possesses a *threshold strength*. Consequently, sensitivity to flaws that would ordinarily cause catastrophic failure at stresses below the threshold strength is eliminated. Furthermore, the potential exists for the complete elimination of the strength variability, hence improving reliability, through

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the creation of nearly deterministic, i.e. single-valued, strengths by increasing the threshold strength above the stresses at which failure normally initiates from intrinsic flaws.

The potential this invention offers for the implementation of brittle materials in high-performance structural applications ranging from high-temperature gas turbine engines to biomedical prosthetic implants is significant. Elimination of the need for designers to rely on probabilistic strength definitions and acceptable failure probabilities will allow for design using conventional engineering methodologies, thereby facilitating the introduction of these materials.

These and other features, aspects, and advantages of the present invention will become better understood with regard to the following detailed description, claims, and accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic representation of the superimposed stress fields used to determine the stress intensity of the arrested crack;

Figure 2 shows the results of the optimization analysis as a plot of t_2/t_1 as a function of the independent variables;

Figure 3 is a plot of the failure strength of the laminated and monolithic specimens with varying starting flaw sizes;

Figure 4 is a scanning electron micrograph of the fracture surface of one of the laminate specimens;

Figure 5 is a plot of the threshold strength vs. magnitude of residual compression within the compressive layers for two different laminate architectures;

Figure 6 is a plot of the threshold strength vs. compressive layer thickness for two different laminate architectures;

Figure 7 is a plot of the threshold strength vs. tensile layer thickness for two different laminate architectures;

Figure 8 is an optical micrograph of replicas made of a crack propagating across the tensile surface of a non-edge cracked laminate;

Figure 9 is a plot of the stress intensity vs. the crack length for a crack propagating through a non-edge cracked compressive layer;

Figure 10 is an optical micrograph of replicas made of a crack propagating across the tensile surface of an edge cracked laminate;

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Figure 11 is a plot of the stress intensity vs. the crack length for a crack propagating through an edge cracked compressive layer;

Figure 12 is a scanning electron micrograph of the fracture surface of a specimen that exhibited edge cracking;

Figure 13 is an optical micrograph showing the bifurcation of the crack within the bulk of an edge cracked specimen, beneath the penetration depth of the edge crack;

Figure 14 is a plot of the stress intensity of a crack propagating through the compressive layer as a function of distance showing the effect of modulus mismatch;

Figure 15 is a plot of the threshold strength vs. amount of m-ZrO_2 for Al_2O_3 laminates with Al_2O_3 / m-ZrO_2 compressive layers;

Figure 16 is a schematic diagram of the composite architecture needed to produce compressive layers within a body formed of hexagonal prisms of one or more materials, separated and bonded together with compressive layers formed of another material; and

Figure 17 is a schematic diagram of the composite architecture needed to produce compressive layers within a body formed of polyhedra of one or more materials, separated and bonded together with compressive layers formed of another material.

DETAILED DESCRIPTION

The hypothesis that multiple, thin compressive layers could lead to a threshold strength in brittle materials had its genesis in an inadvertent observation made by one of us (Antonio Javier Sanchez Herencia) in which a crack was observed to initiate and arrest between two compressive layers during experiments to further understand the phenomena of crack bifurcation, that is, the 90° change in the direction of a crack as it enters and extends along the center line of a compressive layer [5-7]. This observation initiated a fracture mechanics analysis to determine the conditions for crack arrest and subsequent failure, and subsequent experiments to test the analysis [8].

Mechanical Analysis of the Arrest Phenomena and Development of the Threshold Strength Model

A residual, biaxial, compressive stress arises within layers of thickness t_1, either surface or internal, when they are compressed relative to a second set of alternating layers of thickness, t_2. This compression can arise due to a compressive strain mismatch, ε, caused by either a mismatch in the thermal expansion coefficients of the laminae, or by a volume change of either of the laminae through a crystallographic phase transformation or formation of a chemical reaction product. For the specific case of a laminated plate composed of compressive layers (t_1), alternated between tensile layers (t_2), the biaxial stresses in both layers are given by [9]

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$$\sigma_1 = \varepsilon \, E_1' \left(1 + \frac{t_1 E_1'}{t_2 E_2'} \right)^{-1} \quad \text{and} \quad \sigma_2 = -\sigma_1 \frac{t_1}{t_2} \tag{1}$$

where $E_i' = E_i/(1-v_i)$, E is Young's modulus and v is Poisson's ratio. Inspection of the two relations shows that thin compressive layers are desired because when $t_1/t_2 \to 0$ the compressive stress is maximized while the tensile stress diminishes to zero in the thicker layers.

The analysis of the observed arrest phenomena assumes that a pre-existing crack of length 2a spans the thick layer (t_2), sandwiched by the compressive, thin layers of thickness t_1, as shown on the left side of Fig. 1. The magnitude of the biaxial, residual compressive stress within the thin layers is given by σ_c, and the opposing residual tensile stress within the thick layer is given by σ_t. The analysis determines the stress intensity factor for a crack of length 2a when it extends into the compressive layers ($t_2 \le 2a \le t_2 + 2t_1$), under an applied stress, σ_a, parallel to the layers. The stress intensity factor is used to determine the applied stress, σ_{thr}, needed to extend the crack through the compressive layers to produce catastrophic failure.

The stress intensity factor, K, is determined by superimposing the two stress fields shown on the right hand side of Fig. 1, each applied to the same slit crack of length 2a, and each with its own, known stress intensity factor. The first is a tensile stress of magnitude σ_a - σ_c applied to a cracked specimen that does not contain residual stresses. The stress intensity factor for this stress is given by the first term on the right hand side of Eq. 2A. The second is a tensile stress of magnitude σ_c + σ_t, which is only applied across the thick layer, the portion of crack defined by t_2. Its stress intensity factor is given by the second term on the right side of Eq. 2A [10]. The two superimposed stress fields sum to that shown on the left hand side of Fig. 1. The stress intensity factor for the two superimposed stress fields is thus given by

$$K = \left(\sigma_a - \sigma_c \right) \sqrt{\pi a} + \left(\sigma_c + \sigma_t \right) \sqrt{\pi a} \left[\frac{2}{\pi} \sin^{-1} \left(\frac{t_2}{2a} \right) \right] \tag{2A}$$

Substituting $\sigma_t = \sigma_c t_1/t_2$ (from Eq. 1) and rearranging, Eq. 2A better expresses the physical significance of the compressive layers:

$$K = \sigma_a \sqrt{\pi a} + \sigma_c \sqrt{\pi a} \left[\left(1 + \frac{t_1}{t_2} \right) \frac{2}{\pi} \sin^{-1} \left(\frac{t_2}{2a} \right) - 1 \right] \tag{2B}$$

The first term in Eq. 2B is the well know stress intensity factor for a slit crack in an applied tensile field. The second term is always negative and thus reduces the stress intensity factor when the crack extends into the compressive layers. Thus, the compressive layers increase the material's resistance to crack extension.

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Because K decreases as the crack extends into the compressive layers, the maximum stress needed to cause the crack to 'break' through the compressive layers occurs when $2a = t_2 + 2t_1$ and $K = K_c$, the critical stress intensity factor of the thin layer material, a property that describes its intrinsic resistance to crack extension. Substituting these values into Eq. 2B and rearranging, the largest stress needed to extend the crack through the compressive layers is given by

$$\sigma_{thr} = \frac{K_c}{\sqrt{\pi \frac{t_2}{2}\left(1 + \frac{2t_1}{t_2}\right)}} + \sigma_c \left[1 - \left(1 + \frac{t_1}{t_2}\right)\frac{2}{\pi}\sin^{-1}\left(\frac{1}{1 + \frac{2t_1}{t_2}}\right)\right] \tag{3}$$

Equation 3 shows that σ_{thr} increases with the fracture toughness of the thin layer material, K_c, the magnitude of the compressive stress, σ_c, and the thickness of the compressive layer, t_1. One can also show that if the initial crack length in the thick layer is $< t_2$, and the stress needed to extend it is $< \sigma_{thr}$, the crack will be arrested by the compressive layers. However, if the crack is very small and extends at a stress $> \sigma_{thr}$, it will extend though the compressive layers to cause catastrophic failure without being arrested. Thus, Eq. 3 defines a threshold stress, σ_{thr}, below which the laminar body cannot fail when the tensile stress is applied parallel to the layers. This prediction has significant implications in that it offers the opportunity to design structural components with the knowledge that the component will not fail below the specified threshold stress.

It has been shown [11] that the conditions that optimize the threshold strength could be determined by differentiating Eq. 3 with respect to the dependent variable, t_2/t_1. Figure 2 shows the results of this optimization analysis, which plots t_2/t_1 as a function of the independent variables. Optimum threshold strengths are obtained by choosing values for the independent variables that can be practically achieved through material choice and processing constraints, and then determining the dependent variable, t_2/t_1 using Fig. 2. Inspection of Eq. 3 shows that minimization of the layer thicknesses is desirable, as it will lead to higher threshold strengths. However, current processing technology limits the minimum thickness achievable to ~5 μm; this thus serves as the constraint for the optimization. For the given Al_2O_3 / mullite system with 5 μm thick compressive layers, Fig. 2 shows that threshold strength will be optimized at a t_2/t_1 ratio of ~1.6, thus $t_2 = 7.95$ μm and $\sigma_c = 1085$ MPa. Substituting these values into Eq. 3, one finds that a threshold strength of 945 GPa is possible in this particular system, which is nearly as large as the compressive stress, despite that fact that the ratio $t_2/t_1 = 1.6$ produces large tensile stresses in the thicker layers separating the compressive layers.

While this is an impressive result, even higher threshold strengths far in excess of the residual compression may be possible in systems with higher toughness compressive layers. Application of Fig. 2 to a laminate system composed of silicon carbide (SiC) tensile layers and silicon nitride (Si_3N_4, $K_c = 8$ MPa•m$^{1/2}$) compressive layers, with the compressive layers again constrained to 5 μm, yields an estimated threshold strength in excess of 2 GPa, despite the fact that σ_c in this system is only 676 MPa. Strengths of this

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magnitude are rarely if ever seen in polycrystalline ceramics and are closer to those expected for single crystal fibers. Single crystal fibers, however, do not share the flaw tolerance capability that these laminates would.

Experimental Validation of the Model

To test the threshold strength concept and its predicted improvement of flaw tolerance, starting flaws of varying size were introduced into laminar ceramic composite specimens made up of tensile layers composed of aluminum oxide (Al_2O_3) and compressive layers composed of a mixture of mullite (3 Al_2O_3: 2 SiO_2) and Al_2O_3. Residual compressive stresses of ~1.2 GPa were developed in the mullite/alumina layers during cooling from processing temperatures due to the mismatch of the thermal expansion coefficients of the tensile and compressive layers. These specimens were then tested to failure and the strengths were compared to those of unreinforced monolithic specimens with similar sized starting flaws.

Figure 3 shows that, as predicted, the failure stress of the laminates was relatively independent of the initial flaw size, while the strength of the monolithic alumina without the compressive layers was strongly flaw dependent. In addition, the morphology of the fracture surfaces of these specimens (Fig. 4) indicated that not only did the compressive layers arrest the initial propagation of otherwise catastrophic cracking initiating from the starting flaws, but they also allowed the specimens to endure a substantial amount of further loading before failure occurred, despite the presence of cracks that constituted a significant portion of the load-bearing cross-section of the specimen.

Further testing of laminate specimens with varying architectures [12] has validated our theory through independent investigation of the effects of the three most important independent variables in Eq. 3, namely the magnitude of the residual compression, the effect of the compressive layer thickness, and the effect of the tensile layer thickness. Figures 5, 6, and 7 show the effect of these parameters and their close correlation to the strengths predicted through the application of Eq. 3. However, the data also show that the current theory ceases to accurately describe the experimental data when the magnitude of the residual compression is large and/or when the thickness of the compressive layers is large.

Closer inspection of the propagation of the cracking within the compressive layers [13] has revealed that the reason for this discrepancy lies in the presence of two distinctly different crack propagation modes. As seen in Fig. 8, cracking in laminates with thin compressive layers under moderate levels of residual compression is observed to propagate in a stable manner, straight across the compressive layers, exactly as was assumed in the derivation of the current theory. In these cases, close correlation is seen between the predicted and measured threshold strengths, as well as the predicted and measured propagation lengths, as seen in Fig. 9.

However, observations also show that once the residual compression and/or the compressive layer thickness exceed a critical value, edge cracking [14] appears on the

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surface, as seen in Fig. 10, which prevents the further propagation of the crack until failure occurs at much higher loads. Measured failure strengths in these cases generally far exceed those predicted by Eq. 3, while the arrest of the cracking at the edge crack conflicts with predictions of further extension, as seen in Fig 11. Inspection of the fracture surfaces of these specimens (Fig. 12) and observation of the propagation of the crack beneath the penetration depth of the edge crack (Fig. 13) shows that bifurcation of the crack occurs, which begins to explain the discrepancies in measured and predicted threshold strengths. It is intuitively obvious that it will take much more stress to drive two cracks simultaneously at angles severely inclined to the applied tensile stress axis. Crack propagation of this type is not accounted for in the current model, which therefore invalidates its application in these cases.

Further Finite Element Analysis

We are currently exploring the cause of bifurcation with the use of finite element analysis [15]. Early analysis has already shown that the single crack has a higher stress intensity factor relative to two bifurcating cracks. This would be expected since more energy is needed to drive two cracks than one. Thus, the reason does not lie within the magnitude of the strain energy released. Our current using the finite element approach to determine the T stress in front of the crack, as a function of the crack position within the compressive layer, has shown more success. The T stress is not dependent on either the radius vector or the angular position from the crack tip. When the T stress is compressive, the crack is expected to propagate on its symmetry plane; when it is tensile, the crack is expected to deviate onto another plane. Our finite element analysis does show that the T stress becomes a tensile stress when the compressive stress exceeds a given value and therefore begins to explain the cause of bifurcation. Further exploration of this approach is in progress.

Further finite element simulations have also shown that the arrest phenomena are strongly affected by the moduli of the laminae [16]. As can be seen in Fig. 14, the value of the stress intensity factor is decreased as it enters a compressive layer with a lower modulus relative to the thicker, tensile layer. That is, a compressive layer with a lower elastic modulus will further increase the threshold strength because it stores less elastic strain energy relative to a compressive layer made with a material with a higher elastic modulus. Figure 14 also shows that if the elastic modulus of the compressive layer is too large, it may not stop crack extension at all. Thus, the most desired material couples that exhibit a threshold strength should have a compressive layer modulus less than the tensile layer modulus.

Developing Compressive Stresses via a Structural Phase Transformation

Although most of the experiments to test the initial model were performed with alumina and mullite/alumina layered materials that used the differential thermal contraction during cooling to develop the compressive stresses, we have also initiated a study [17] that uses a structural phase transformation to induce the biaxial compressive stresses. In the current case, we use the tetragonal to monoclinic phase transformation of

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unstabilized ZrO_s to induce the compressive stresses. In this case, the thick (tensile) layers were composed of Al_2O_3, and the thinner compressive layers were formed of a mixture of unstabilized ZrO_2 and Al_2O_3. It was expected that the magnitude of compressive stresses would be controlled by the fraction of unstabilized ZrO_2 in the mixed layer, due to its molar volume increase during the structural phase transformation that occurs during cooling.

Figure 15 reports the threshold strength as a function of the fraction of unstabilized ZrO_2 in the mixed layer. As shown, the threshold strength is high, but does not change with the volume fraction of ZrO_2 for fractions greater than 0.30 volume fraction of the unstabilized ZrO_2. This system is currently under study to understand the relation between the transformation and compressive stresses that arise during the biaxially constrained phase transformation.

Extension of the Threshold Concept to More Isotropic 3-Dimensional Architectures

While the majority of the work undertaken thus far has focused on 2-dimensional laminated materials, it is immediately apparent that these anisotropic architectures will be unable to yield a threshold strength in conditions other than the highly simplified ones described thus far (i.e. loading direction parallel to the laminar plane, driving cracks oriented normal to the layers). In order for the threshold concept to be more versatile and robust, higher dimensionality composite architectures must be developed that allow for more isotropic arrest behavior.

As shown in Figs. 16 and 17, we have discovered two different architectures where the compressive layer will stop cracks that extend in any direction. In the first [18], rods of one material are coated with a second material that will produced the compressive stresses. After bundling the coated rods, they are compressed and densified at high temperature to form a solid body that approximates an array of hexagonal prisms, with separated compressive layers on all sides as shown in cross section. Cracks that extend either along the axis or across the diameter of the hexagonal prisms will be stopped by the compressive layers that mutually bond the hexagonal prisms into a solid body.

In the second method [19], spheres of a material are coated with a second material that will form the compressive layer. As shown in Fig. 17, the spheres are mutually deformed by an applied isostatic pressure that converts the spheres into polyhedra that are separated by layers of a material that will form compressive layers. This array of polyhedra is then densified with a high temperature heat treatment to form a dense, solid body composed of polyhedra of one material surrounded and bonded together by compressive layers of a second material. It is obvious that cracks that extend in any direction within the material forming the polyhedra will be stopped by the compressive layers.

Although the foregoing invention has been described in some detail by way of illustration and example for purposes of clarity and understanding, various modifications

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and changes which are within the knowledge of those skilled in the art are considered to fall within the scope of the claims.

Supplemental Descriptive Material

The following list contains descriptive information that elaborates upon and clarifies the claims set forth below:

Clarification of Claim 1: All other factors being equal, the smaller the separation distance between compressive regions, the higher the threshold strength. The threshold strength is optimized when the distance separating the regions of the material(s) that do(es) not contain compressive stresses is between 0.2 and 0.01 times the dimension of the material(s) that do(es) not contain the compressive stresses, as measured from the interface between the materials. All else being equal, the larger the compressive stress, the larger the threshold stress. Compressive stresses in the range of 500 MPa to 5000 MPa are desired.

Clarification of Claim 2: The layers of materials containing biaxial, residual, compressive stresses are known as compressive layers.

Clarification of Claim 9: Examples of two materials chosen from this list would be alumina and zirconia, where the compressive stresses would arise in the alumina due to its lower thermal contraction coefficient; another example would be silicon nitride and silicon carbide, where the compressive stresses would arise in the silicon nitride due to its lower thermal contraction coefficient. A third example would be alumina and mullite, where the compressive stresses would arise in the mullite during cooling due to its lower thermal contraction coefficient.

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THE CLAIMS

1. A composite comprised of at least two materials that exhibits a threshold strength in which

    the materials are strongly bonded together;

    residual, compressive stresses exist in only one of the materials;

    the material containing the compressive stresses stops cracks extending from the other material(s) at an applied tensile stress less than the threshold strength;

    each material has a different chemical composition relative to the other(s);

    the material(s) that do(es) not contain the compressive stresses possess(es) the largest volume fraction in the composite;

    the material containing compressive stresses separates and exists between the regions of the material(s) that do(es) not contain compressive stresses;

    the dimension of materials that do not contain compressive stresses, measured perpendicular to the interface between the materials, is smaller than 1000 microns;

    the thickness of the regions that contain the compressive stresses is between 1 times and 0.001 times the dimension of the material(s) that do(es) not contain the compressive stresses, as measured from the interface between the two materials; and

    the compressive stress is greater than 100 MPa.

2. A composite as in claim 1, in which layers of one material are separated by alternating layers of one or more other materials; and in which all sets of layers are bonded together; and in which one of the alternating materials contains biaxial, residual, compressive stresses; and in which, at applied tensile stresses less than a threshold value, cracks that extend into the compressive layers are stopped and cannot cause catastrophic failure.

3. A composite as in claim 1, in which prismatic cylinders of one or more materials are separated and bonded to another material that contains residual compressive stresses; and in which, at applied tensile stresses less than a threshold value, cracks that attempt to propagate from the prismatic cylinders into the compressive layers are stopped and cannot cause catastrophic failure.

4. A composite as in claim 1, in which polyhedra of one or more materials are separated and bonded to another material that contains residual compressive stresses; and in which, at applied tensile stresses less than a threshold value, cracks that attempt to propagate from the polyhedra into the compressive layers that surround the polyhedra are stopped and cannot cause catastrophic failure.

5. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise during cooling from a processing temperature to room temperature, caused by the differential strain induced by the differential thermal contraction coefficients of the different materials used to form the composite.

6. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise during cooling from a processing temperature to room temperature, caused by a volume expansion of one of the materials that undergoes a crystallographic phase transformation during cooling.

7. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise when two materials react together to form a third material, and this reaction is accompanied by an increase in molar volume which is constrained by a fourth material that does not take part in the reaction.

8. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise when two materials react together to form a third material; and in which this reaction is accompanied by an increase in molar volume which is constrained by a fourth material that does not take part in the reaction; and in which the two materials that react together are silica (SiO_2) and alumina (Al_2O_3), which form mullite (3 Al_2O_3 : 2 SiO_2), the third material; and in which this reaction produces a molar volume increase which, when constrained by a fourth material, such as but not limited to alumina (Al_2O_3), will produce a compressive stress.

9. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise during cooling from a processing temperature to room temperature, caused by the differential strain induced by the differential thermal contraction coefficients of the different materials used to form the composite; and in which the materials are chosen from a list that includes at least two materials that do not react together to form a third material, but have differential thermal contraction coefficients such that compressive stresses would arise in one of the materials during cooling from a processing temperature, this list including, but not limited to, alumina (Al_2O_3), zirconia (ZrO_2), mullite (3 Al_2O_3 : 2 SiO_2), silicon nitride (Si_3N_3), silicon carbide (SiC), and titania (TiO_2).

10. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise during cooling from a processing temperature to room temperature, caused by the differential strain induced by the differential thermal contraction coefficients of the different materials used to form the composite; and in which the materials are chosen from a list that includes at least two materials that do not react together to

form a third material, but have differential thermal contraction coefficients such that compressive stresses would arise in one of the materials during cooling from a processing temperature, this list including, but not limited to, alumina (Al_2O_3), zirconia (ZrO_2), mullite ($3 \ Al_2O_3 : 2 \ SiO_2$), silicon nitride (Si_3N_3), silicon carbide (SiC), and titania (TiO_2); and in which the material(s) that do(es) not contain the compressive stresses do(es) contain another material that would impart an additional property important to mechanical strength and thus optimize factors that affect threshold strength that include, but are not limited to, controlling grain growth, changing the coefficient of thermal contraction, and changing the elastic modulus.

11. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise during cooling from a processing temperature to room temperature, caused by the differential strain induced by the differential thermal contraction coefficients of the different materials used to form the composite; and in which the materials are chosen from a list that includes at least two materials that do not react together to form a third material, but have differential thermal contraction coefficients such that compressive stresses would arise in one of the materials during cooling from a processing temperature, this list including, but not limited to, alumina (Al_2O_3), zirconia (ZrO_2), mullite ($3 \ Al_2O_3 : 2 \ SiO_2$), silicon nitride (Si_3N_3), silicon carbide (SiC), and titania (TiO_2); and in which the material that contains the compressive stresses also can contain another material that would impart an additional property important to mechanical strength and thus optimize factors that affect the threshold strength that include, but are not limited to, controlling grain growth, changing the coefficient of thermal contraction, and changing the elastic modulus.

12. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise during cooling from a processing temperature to room temperature, caused by the differential strain induced by the differential thermal contraction coefficients of the different materials used to form the composite; and in which one or more of the materials is an amorphous material, without long range atomic order.

13. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise during cooling from a processing temperature to room temperature, caused by the differential strain induced by the differential thermal contraction coefficients of the different materials used to form the composite; and in which the materials are not ceramics such as oxides, carbides, nitrides, and borides, but are materials chosen from a group of materials known as polymers, where one polymer would have a lower thermal contraction coefficient relative to the other polymer(s).

14. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise during cooling from a processing temperature to room temperature, caused by the differential strain induced by the differential thermal contraction coefficients of the different materials used to form the composite; and in which the materials are not either ceramics or polymers, but are at least two materials chosen from a

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group of materials known as metals, where one metal would have a lower thermal contraction coefficient relative to the other metal(s).

15. A composite as in claims 1, 2, 3, or 4, in which the compressive stresses arise during cooling from a processing temperature to room temperature, caused by a volume expansion of one of the materials that undergoes a crystallographic phase transformation during cooling; and in which the material that exhibits a volume increase due to a structural phase transformation is zirconia with the tetragonal structure at high temperature, which transforms at a temperature below approximately 1100 °C to its monoclinic structure with a volume increase of approximately 3 %; and in which other materials including, but not limited to, alumina (Al_2O_3) and mullite ($2SiO_2 \cdot 3Al_2O_3$) can be used as the other material(s) in the composite, which would partially constrain the volume increase of the zirconia material and thus produce the desired compressive stresses in the transformed zirconia material.

16. A method of processing the composites as in claims 1, 2, 3, or 4, using powder of the materials which are heated to a temperature where the materials are made dense by sintering.

17. A method of processing the composite as in claims 1 or 2, comprising the following steps: each of the materials is first formed as powder sheets; the sheets are then alternately stacked together and pressed together into contact; and then the sheets are heated to a temperature that would cause the densification of the materials.

18. A method of processing the composite as in claims 1 or 3, comprising the following steps: one or more materials are first formed from a powder as cylindrical rods; the cylindrical rods are then coated with another material in which compressive stresses will arise during processing; the coated rods are pressed together to deform them into prismatic cylinders that are separated from each other by the coating material; and, after this pressing operation, the composite structure is then made dense by heating to a high temperature to induce sintering.

19. A method of processing the composite as in claims 1 or 4, comprising the following steps: one or more materials are first formed from a powder as spheres; the spheres are then coated with another material in which the compressive stresses will arise during processing; the coated spheres are then pressed together to deform them into polyhedra that are separated by the coating material; and the powder composite is then heated to a temperature where the powders densify to form a dense composite material.

20. A method of processing the composite as in claims 1 or 2, in which the materials are already dense materials that are stacked together and bonded together with an applied pressure.

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21. A method of processing the composite as in claims 1, 3, or 4, in which the materials that do not contain the compressive stresses are separately made with the described shape and then bonded together with the material that will contain the compressive stress.

ABSTRACT

Reliable, flaw-tolerant brittle materials are produced by incorporating layers under residual compression on the surface and throughout the bulk of the material that act to trap and contain the propagation of otherwise catastrophic cracking. The residual compression within these layers acts to reduce the stress intensity of the cracks, thereby causing them to arrest until further loading is provided. This highly desirable stable, subcritical crack growth mode persists with increased loading until the applied stress is large enough to drive the crack completely through compressive region, after which failure occurs. The exact level of stress needed to accomplish this is dictated by the architectural design of the compressive layers such that the material can be designed to have any minimum strength desired, within the limits of the materials system used. This results in a truncation of the strength distribution, such that there is virtually zero probability of failure below this minimum value, i.e. the material possesses a *threshold strength*. Consequently, sensitivity to flaws that would ordinarily cause catastrophic failure at stresses below the threshold strength is eliminated. Furthermore, the potential exists for the complete elimination of the strength variability, hence improving reliability, through the creation of nearly deterministic, i.e. single-valued, strengths by increasing the threshold strength above the stresses at which failure normally initiates from intrinsic flaws.

17
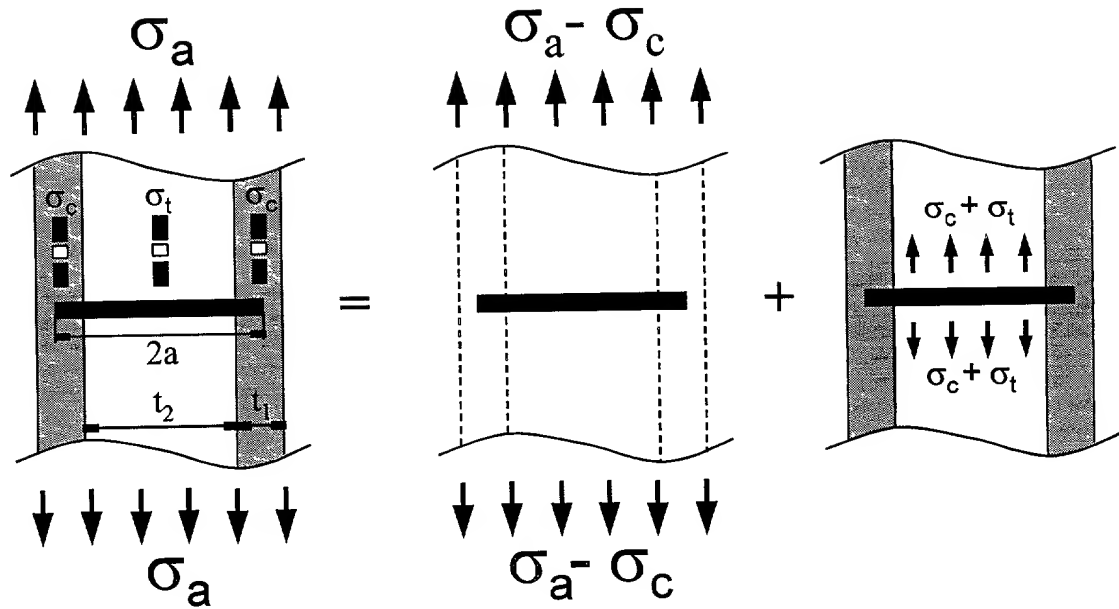
Fig. 1 Schematic representation of the superimposed stress fields used to determine the stress intensity factor of the arrested crack. The left hand side shows a laminar ceramic composed of thin layers (t_1) subject to residual compressive stresses (σ_c) and thick layers (t_2) subject to tensile stresses (σ_t) all subjected to an applied tensile stress (σ_a). This laminate contains a slit crack of length 2a that extends into the compressive layers. The stresses shown on the left can be produced by the superposition of the two stress states shown on the right.

$$\frac{t_2}{t_1}$$

Optimal results for
$$K_c/(-\varepsilon E')\sqrt{\pi t_1/2} \leq 0.883$$

$$t_1 = t_2$$
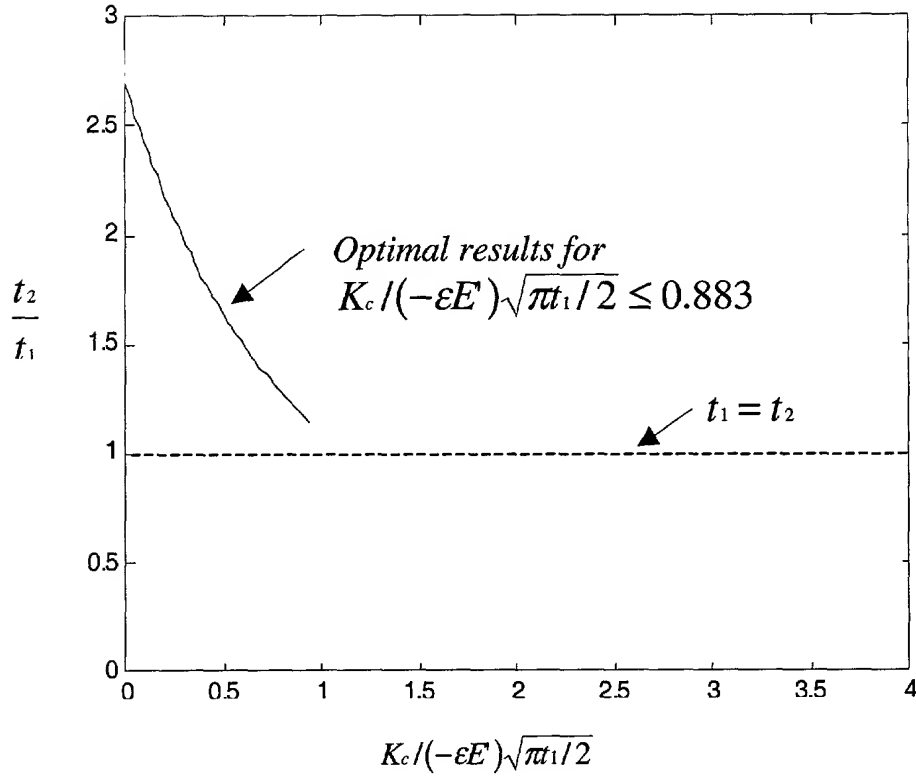
$$K_c/(-\varepsilon E')\sqrt{\pi t_1/2}$$

Fig. 2 Plot of the layer thickness ratio, t_2/t_1, that optimizes the threshold strength for a given fixed compressive layer thickness, t_1. When $K_c/(-\varepsilon E')\sqrt{\pi t_1/2} \leq 1$, the residual compression term dominates, therefore threshold strength is optimized when the tensile layer is a few times thicker than the compressive layer. When $K_c/(-\varepsilon E')\sqrt{\pi t_1/2} \geq 1$, the toughness term dominates, therefore threshold strength is optimized when the layer thicknesses are equal and chosen to be as thin as practically possible, as indicated by the dashed line.
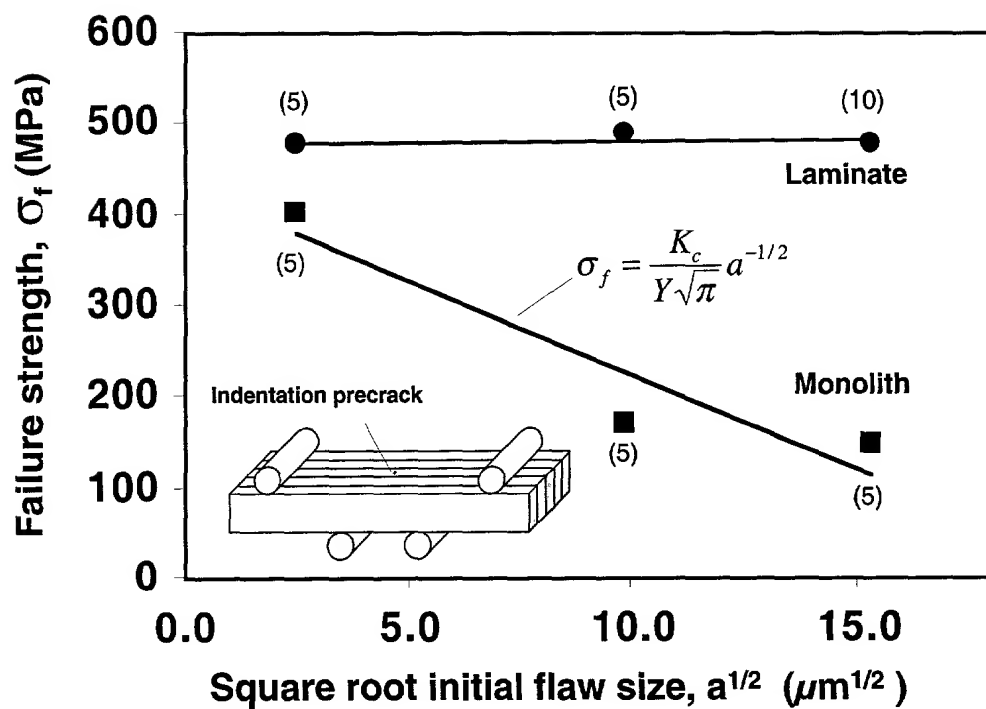
Fig. 3 Plot of the failure strength vs. the square root of the initial flaw size for both laminated and monolithic specimens, tested in the configuration shown in the inset. Numbers in parentheses indicate the number of specimens tested for that data point; the standard deviation for each was ≤ 5%.
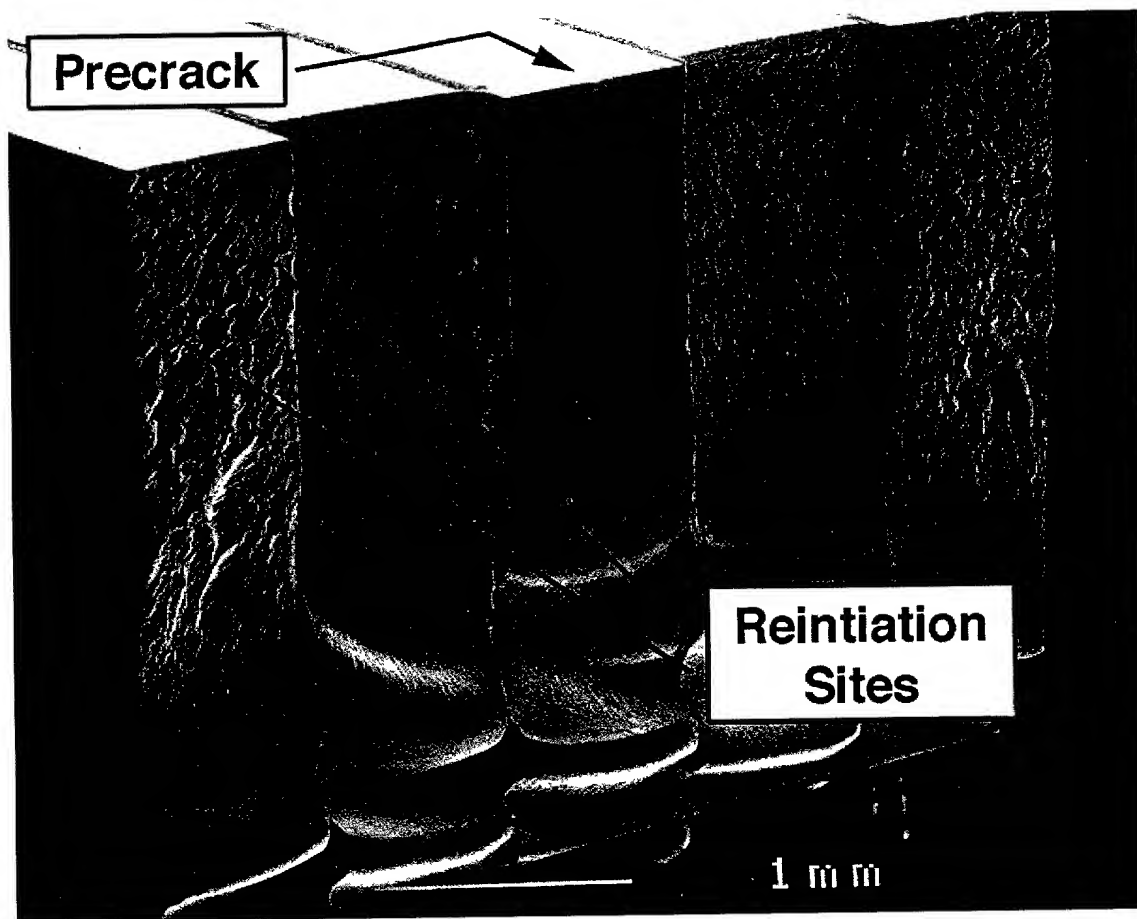
Fig. 4 A scanning electron micrograph of a typical fracture surface where fracture initiated, then arrested, in the central layer from the indentation crack. The top surface was the tensile face of the specimen.
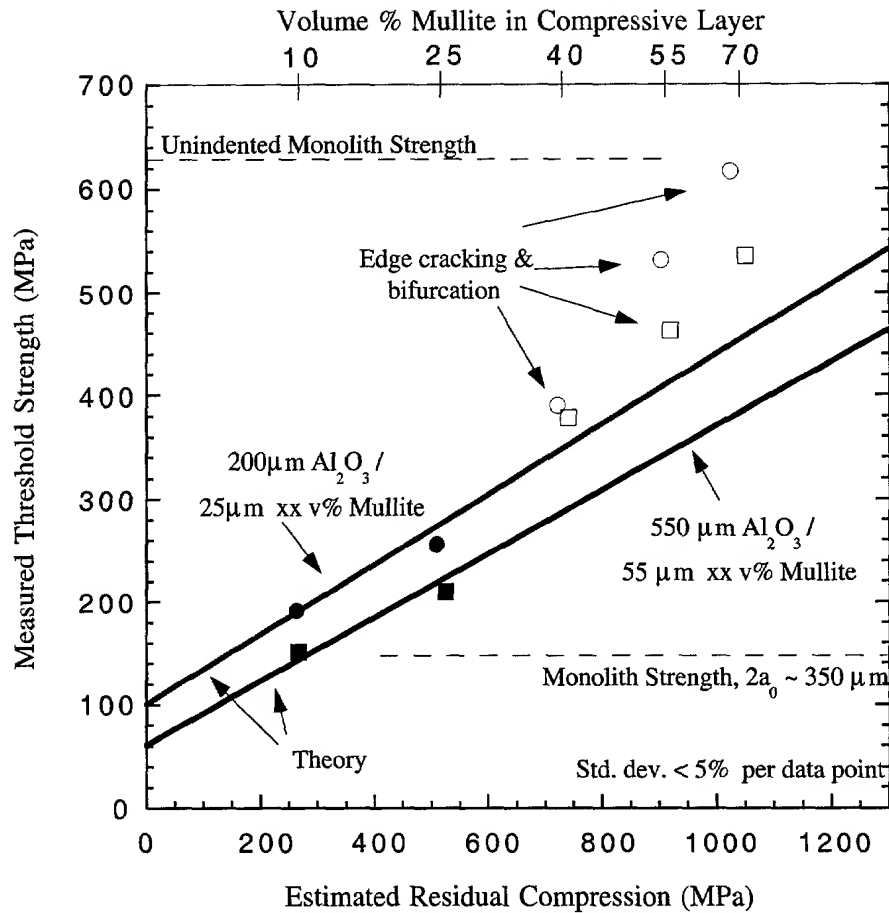
Fig. 5 Experimental data to determine the effect of residual, biaxial compressive stress on threshold strength. Solid lines represent theory for the two different composites architectures (both have similar layer thickness ratios for each composition, therefore similar residual stress levels, but different overall thicknesses). Composites that agree well with theory have cracks that extend straight through compressive layer as modeled by theory. Composites that have a higher threshold strength relative to theory have cracks that bifurcate within the compressive layers.

450
Unindented Monolith Strength = 629 ± 118 MPa

Measured Threshold Strength (MPa)

400

350

Edge cracking &
bifurcation

300

500 μm Al$_2$O$_3$ / xx μm 40 v% Mullite
(σ_c = 746 MPa)

Theory

250

200

500 μm Al$_2$O$_3$ / xx μm 25 v% Mullite
(σ_c = 529 MPa)

150

Monolith Strength, 2a$_0$ ~ 350 μm

100

Std. dev. < 5% per data point

50

0   20   40   60   80   100

Compressive Layer Thickness, t$_l$ (μm)
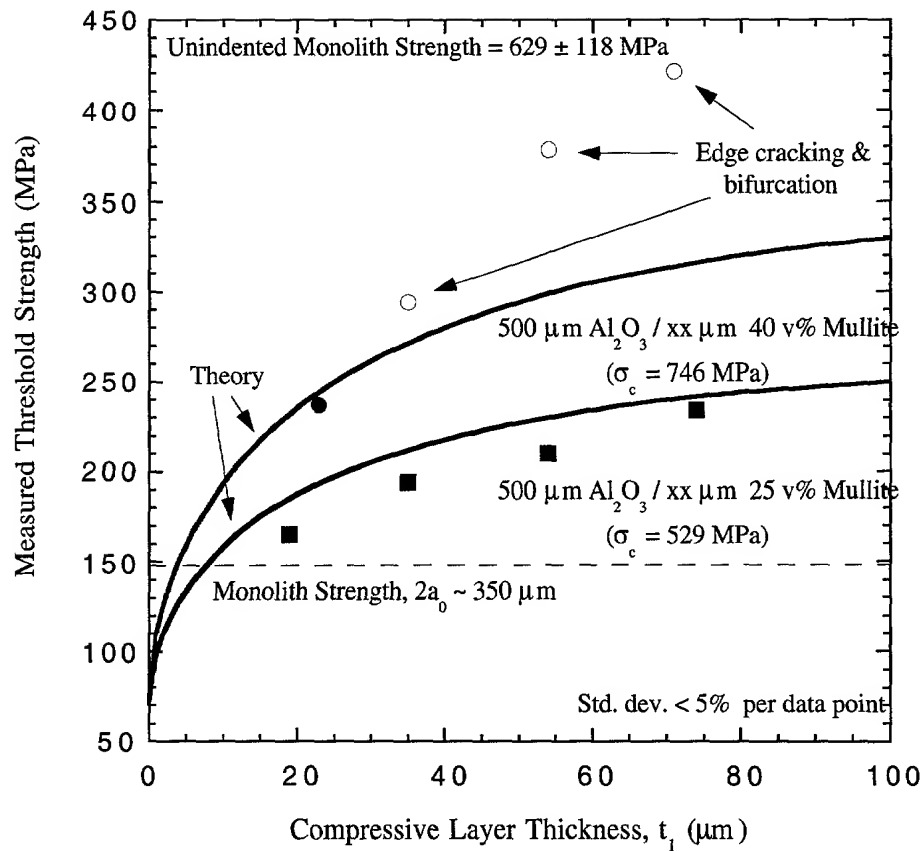
Fig. 6 Experimental data used to determine the effect of compressive layer thickness on
threshold strength. Solid lines represent theory for the two different composites
architectures (both have similar layer thickness ratios but different compositions for each
compressive layer thickness, therefore different residual stress levels). Composites that
agree well with theory have cracks that extend straight through compressive layer as
model by theory. Composites that have a higher threshold strength relative to theory
have cracks that bifurcate within the compressive layers.
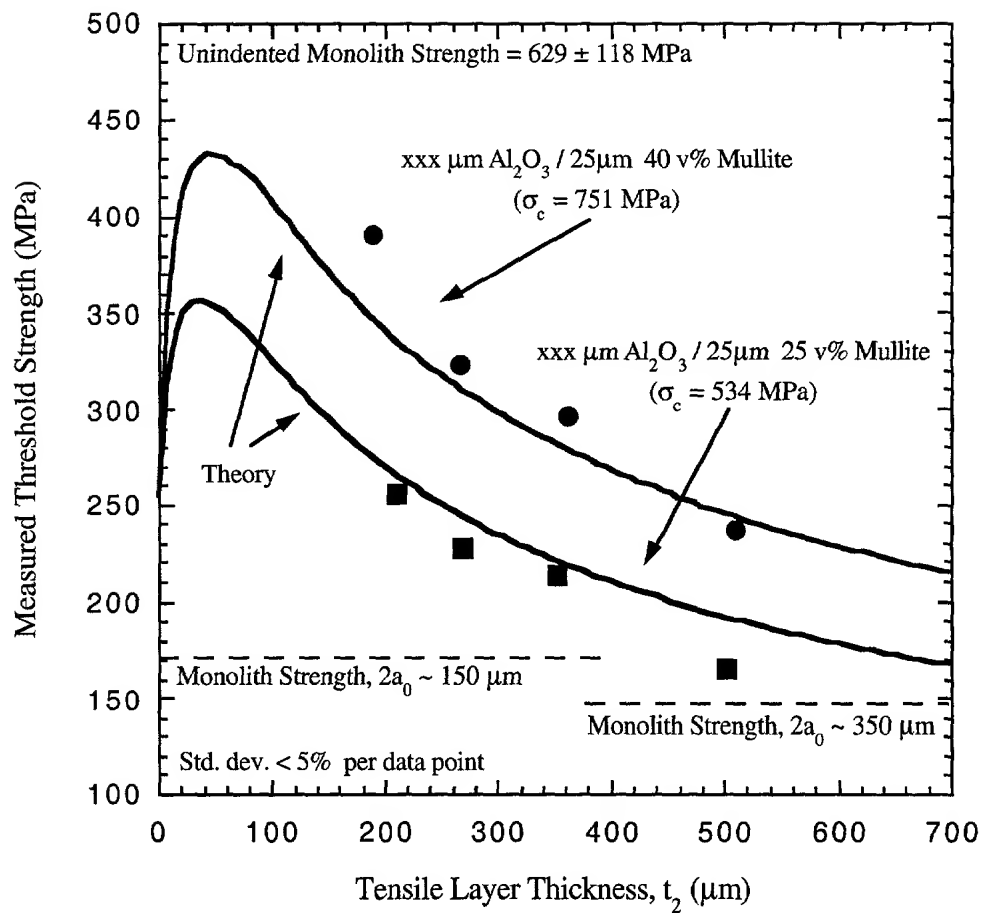
Fig. 7 Experimental data used to determine the effect of tensile layer thickness on threshold strength. Solid lines represent theory for the two different composites architectures (both have similar layer thickness ratios but different compositions for each tensile layer thickness, therefore different residual stress levels). Composites that agree well with theory have cracks that extend straight through compressive layer as model by theory.
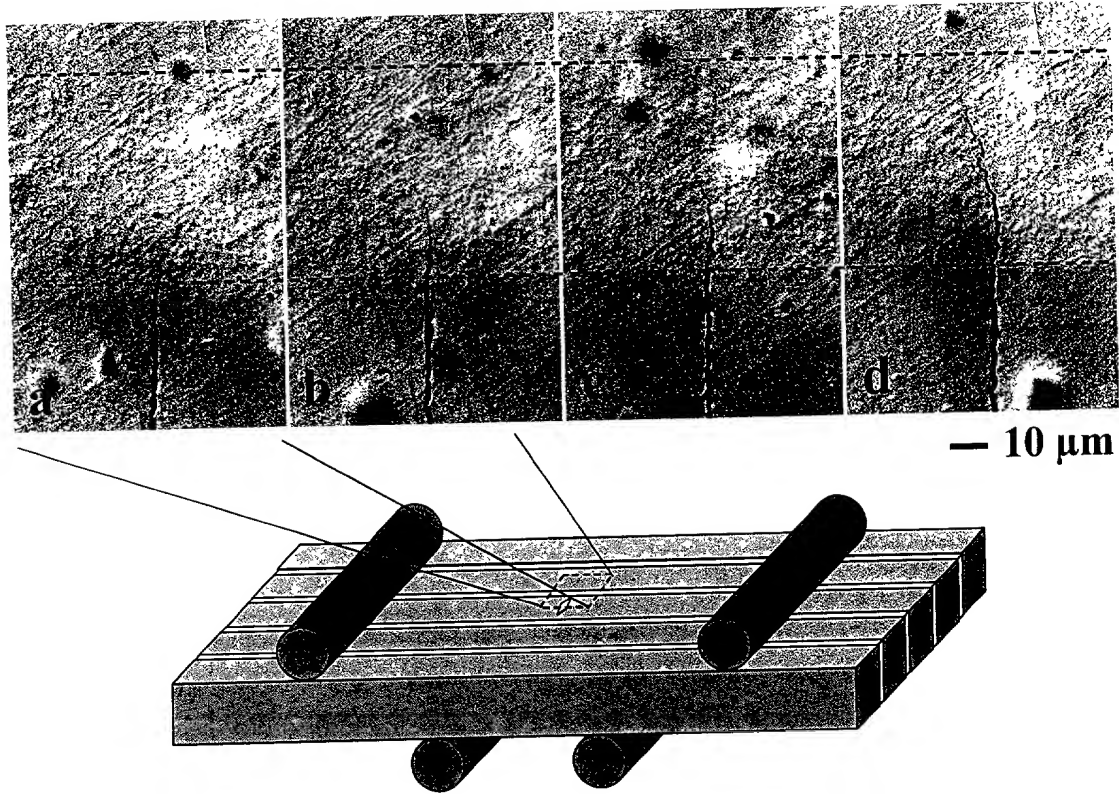
Fig. 8 Schematic of the transverse 4-point flexure configuration and optical micrographs of cellulose acetate replicas of the arrested crack within one of the compressive layers of one of the specimens with compressive layers formed with 0.25 volume fraction of mullite taken during loading at applied stresses of: a) 112 MPa; b) 140 MPa; c) 168 MPa; and d) 195 MPa. The specimen failed soon after at an applied stress of 208 MPa. Crack penetration depths within the compressive layers of 10,17.5, 30, and 47.5 μm, respectively, were measured from the micrographs. Note: Dashed lines were added to indicate the boundaries of the compressive layer.
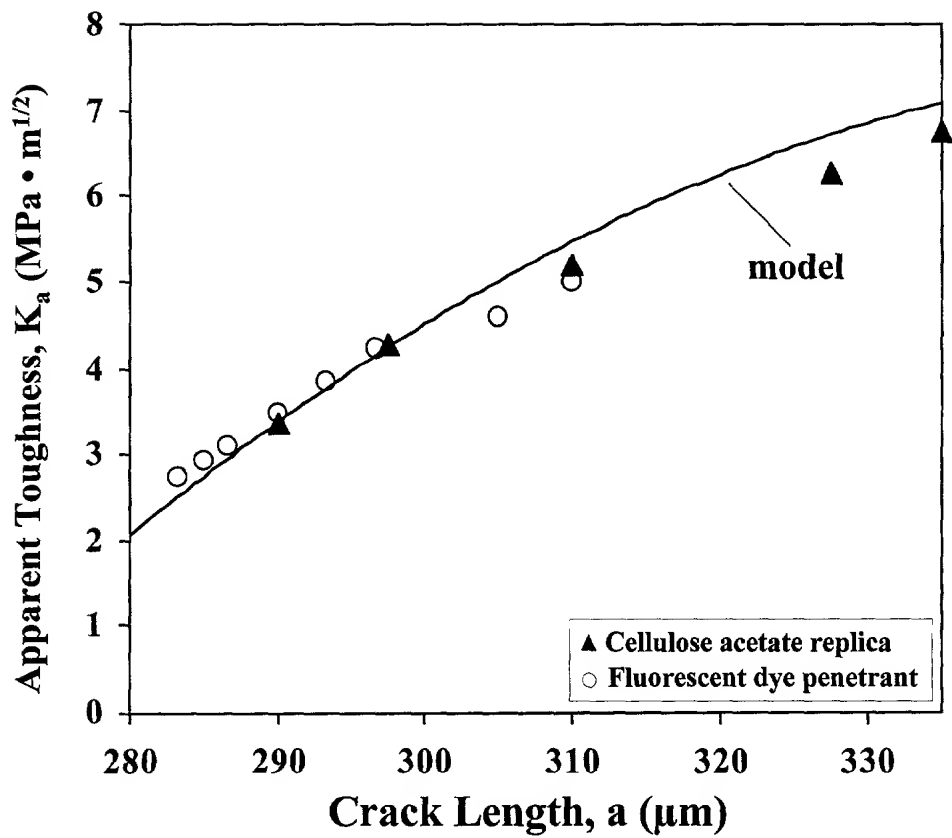
Fig. 9 Plot of predicted vs. observed *apparent toughnesses*, K_a, for cracks propagating through compressive layers formed of 0.25 volume fraction of mullite. Solid line represents model prediction. Crack growth was observed by either cellulose acetate replication or fluorescent dye penetrant.
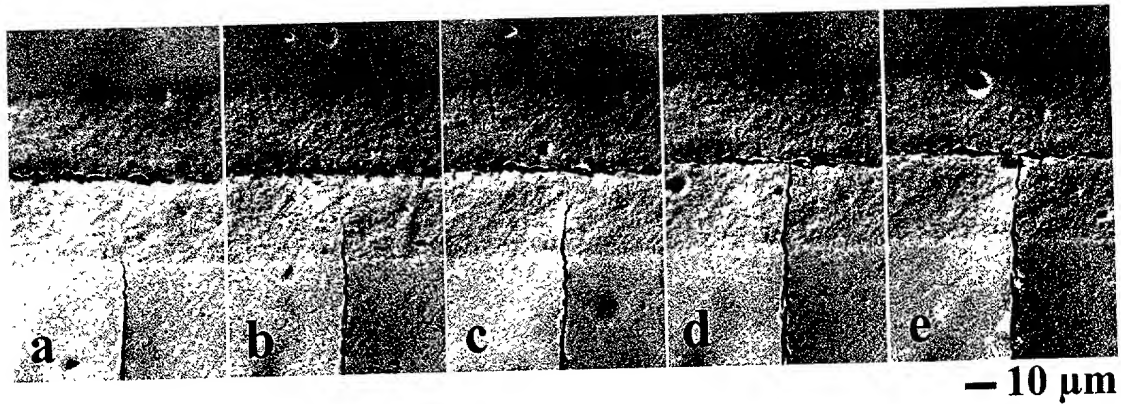
Fig. 10 Optical micrographs of cellulose acetate replicas of the arrested crack within one of the compressive layers of one of the specimens with compressive layers formed with 0.55 volume fraction of mullite taken during loading at applied stresses of: a) 100 MPa; b) 151 MPa; c) 201 MPa; d) 251 MPa; and e) 301 MPa. Crack lengths of 5, 10, and 20 μm, respectively, were measured before the crack reached the edge crack (25 μm into the compressive layer) after which no further propagation was observed until failure occurred at an applied stress of 389 MPa. Note: Cracking observed along the midplane of the compressive layer occurred before loading and is the edge crack that formed on the new free surface revealed by grinding and polishing.
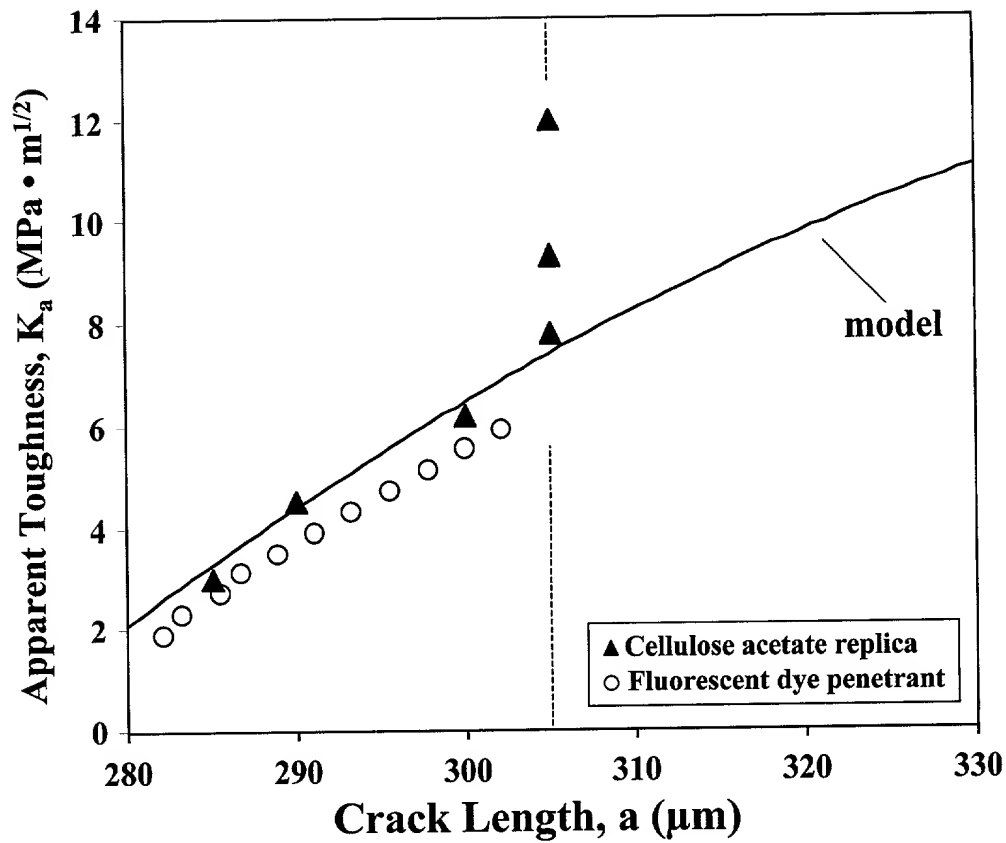
Fig. 11 Plot of predicted vs. observed *apparent toughnesses, K_a* for cracks propagating through compressive layers formed of 0.55 volume fraction of mullite. Solid line represents model prediction. Crack growth was observed by either cellulose acetate replication or fluorescent dye penetrant. The broken vertical dashed line on plot indicates the position of the edge crack in the compressive layer.
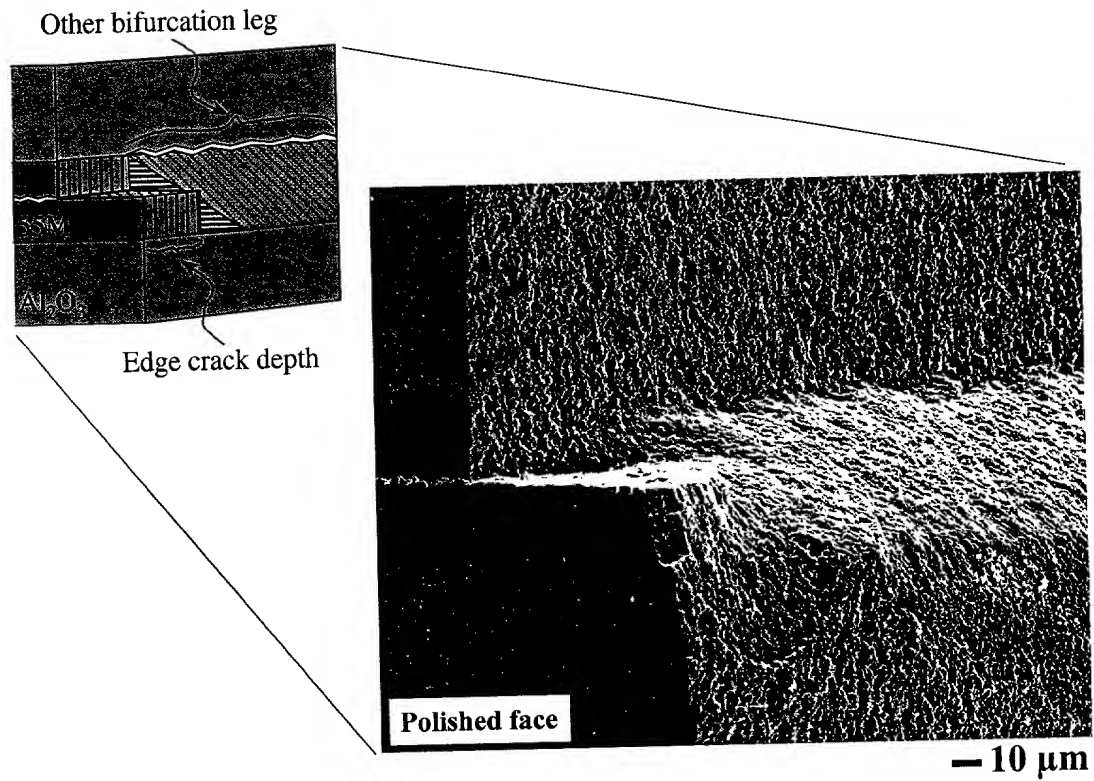
Fig. 12 Scanning electron micrograph and schematic of the fracture surface of one of the specimens with 0.55 volume fraction mullite compressive layers in the region near the tensile face of the bar (smooth area on left of picture) showing the transition between the cracking observed on the surface and the bifurcation that occurs within the bulk, beneath the penetration depth of the edge crack.
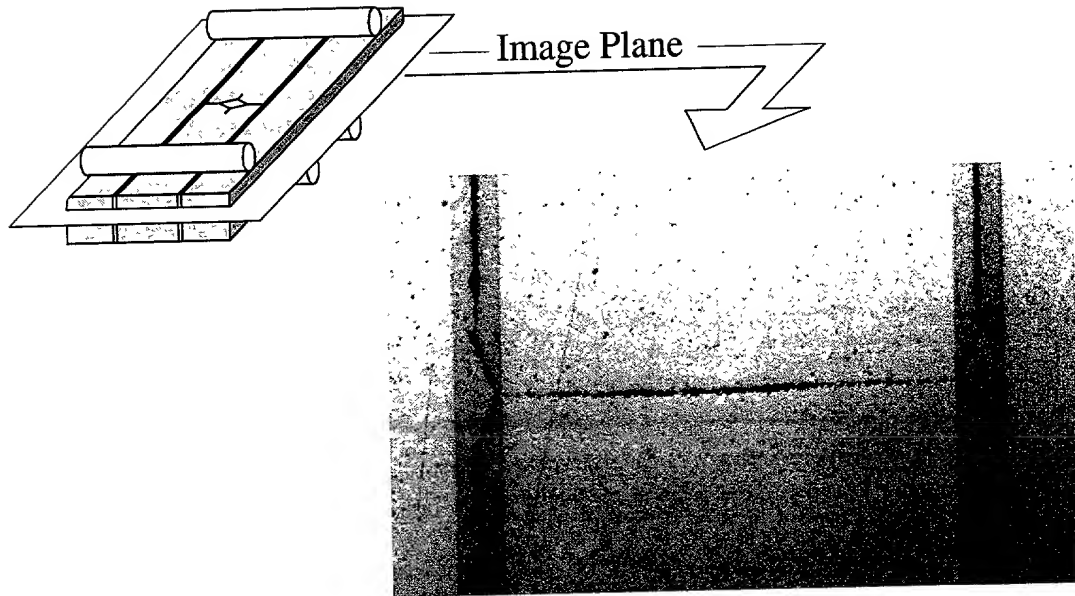
Fig. 13 Optical micrograph of the bifurcation of the crack beneath the tensile surface of a specimen with 0.55 volume fraction compressive layers revealed by grinding down to just below the penetration depth of the edge crack (~30 μm). Note: Observed edge crack reformed on the new free surface upon grinding, as is evidenced by its absence between the branches of the bifurcated crack where the tensile surface stresses are relieved.
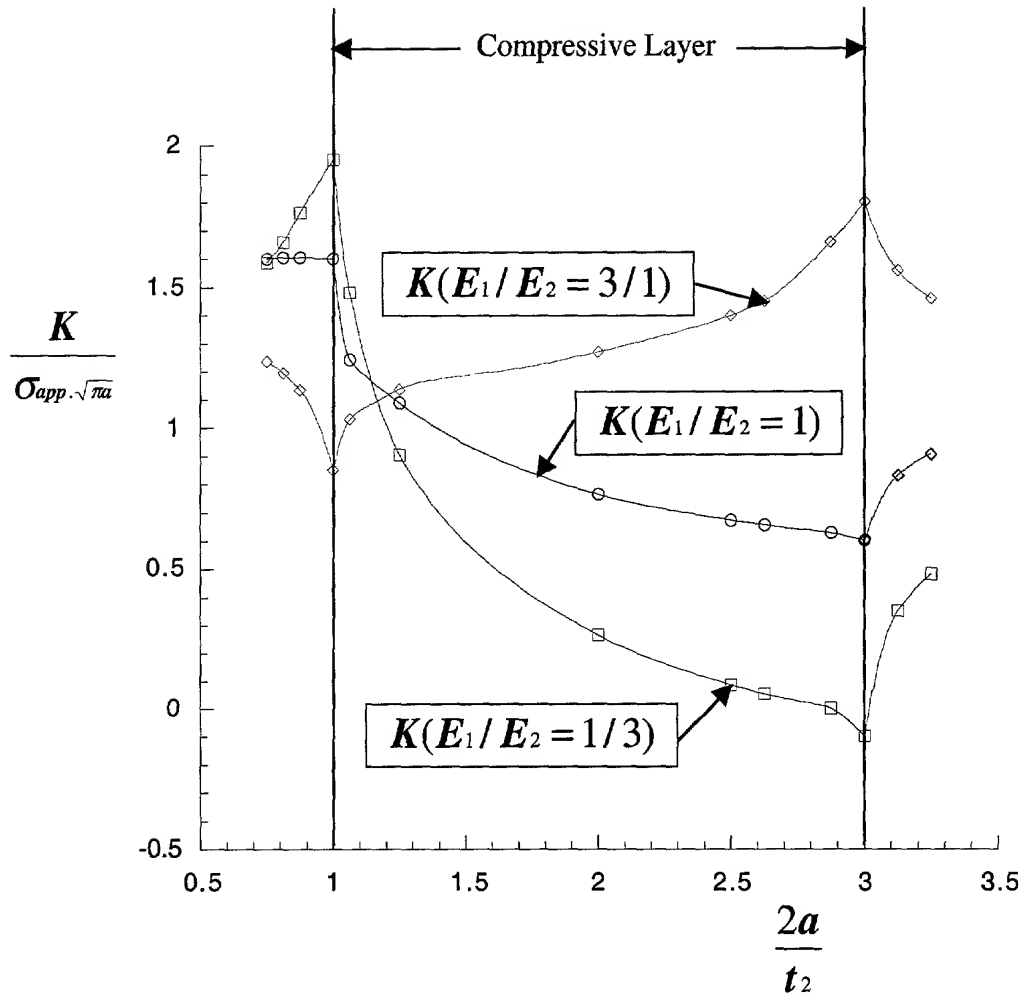
Fig. 14 Results of finite element analysis for crack extension through a compressive layer with three different elastic moduli ratios. For the three cases shown, largest resistance to crack propagation is obtained when $E_1/E_2 = 1/3$, that is, when the elastic modulus of compressive layer is lower than thicker, tensile layer.
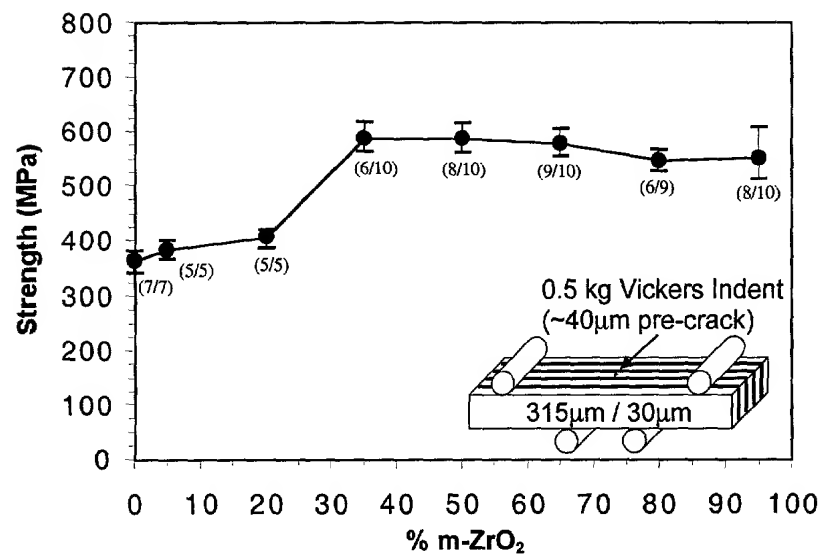
Fig. 15 Threshold strength vs. vol% monoclinic (unstabilized) zirconia in compressive layer formed with mixture of m-zirconia + alumina to change compressive stress arising during phase transformation.

coated rods

coated rods packed together

**coated rods pressed together,
all are now in conact and
separated by compressive layers**
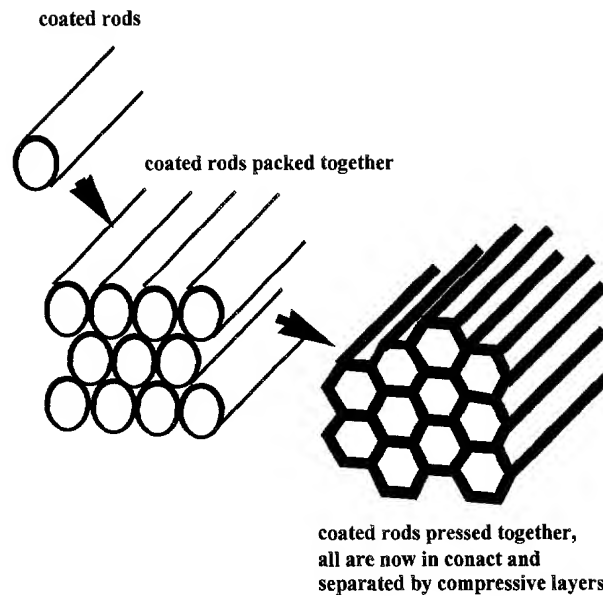
Fig. 16 Composite architecture developed to produce compressive layers within body formed of hexagonal prisms of one or more materials, separated by and bonded together with compressive layers formed of another material. Figure illustrates that this architecture is produced by coating rods formed of one or more materials with another material that will produced a compressive stress, stacking them in a hexagonal array, pressing them together, and then heat treating the array at high temperatures to bond the prisms together and produced densification.

coated spheres in point contact

contact area grows
with increasing
applied pressure

contact area fully grown

Fig. 17 Composite architecture developed to produce compressive layers within body formed of polyhedra of one or more materials, separated and bonded together with compressive layers formed of another material. Figure illustrates that this architecture is produced by coating spheres formed of one or more materials with another material that will produced a compressive stress, packing them together, pressing them together to convert the point contact into a complete area contact (follow arrows from top to bottom, and then heat treating the array at high temperatures to bond the polyhedra together and produce densification.)

DECLARATION AND PETITION

As the below named inventor I hereby declare that:

My residence, post office address and citizenship is as stated below next to my name.

I believe that I am an original, first inventor of the subject matter which is claimed and for which a patent is sought on the invention entitled METHOD FOR IMPROVING THE RELIABILITY OF BRITTLE MATERIALS THROUGH THE CREATION OF A THRESHOLD STRENGTH, the specification of which is attached hereto.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, Section 1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed: None.

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s), and Section 119(e) of any United States provisional application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Section 112, we acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Section 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application: None.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Wherefore I pray that Letters Patent be granted to me for the invention or discovery described and claimed in the foregoing specification and claims, and I hereby subscribe my name to the foregoing specification and claims, declaration and petition.

FREDERICK F. LANGE

Inventor's Signature: _Frederick F. Lange_

Date: ___Sept 26, 2000___

Residence: 1175 Orchid Drive, Santa Barbara, California 93111

Citizenship: United States of America

Post Office Address: Same as Residence Address

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MASA P. RAO

Inventor's Signature: _____

Date: ___9/26/2000___

Residence: 6504 Madrid Road, Apartment G, Isla Vista, California 93117
Citizenship: United States of America
Post Office Address: Same as Residence Address

DECLARATION AND PETITION

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I believe that I am an original, first inventor of the subject matter which is claimed and for which a patent is sought on the invention entitled METHOD FOR IMPROVING THE RELIABILITY OF BRITTLE MATERIALS THROUGH THE CREATION OF A THRESHOLD STRENGTH, the specification of which is attached hereto.

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Wherefore I pray that Letters Patent be granted to me for the invention or discovery described and claimed in the foregoing specification and claims, and I hereby subscribe my name to the foregoing specification and claims, declaration and petition.

ANTONIO JAVIER SANCHEZ HERENCIA

Inventor's Signature: _____

Date: _____27th of SEPTEMBER of 2000_____

Residence: C/ Juan de Austria, 13, 3-C, 28010 Madrid, Spain

Citizenship: Spain

Post Office Address: Same as Residence Address

APPLICANTS: Frederick F. Lange, Masa P. Rao, and   )
                      Antonio Javier Sanchez Herencia   )
)

SERIAL NO.:   )
)

FILED: September 29, 2000   )
)

FOR: METHOD FOR IMPROVING THE   )
                        RELIABILITY OF BRITTLE MATERIALS)
                        THROUGH THE CREATION OF A   )
                        THRESHOLD STRENGTH   )
)

Examiner:)
)

Docket: 1999-385-1   )
)

POWER OF ATTORNEY BY ASSIGNEE
AND EXCLUSION OF INVENTOR UNDER RULE 37 C.F.R. 3.71

Office of Technology Transfer
1111 Franklin Street, 5th Floor
Oakland, California 94607-5200

Assistant Commissioner for Patents
Washington, D.C. 20231

Dear Assistant Commissioner:

The undersigned assignee of the entire interest in the above-identified subject application hereby appoints Tim W. Wan, Registration Number 45,421, as its attorney to prosecute this application and to transact all business in the United States Patent and Trademark Office connected therewith, said appointment to be to the exclusion of the inventors and their attorney in accordance with the provisions of 37 C.F.R. 3.71.

Please direct all telephone calls to Tim W. Wan at (510) 587-6071 and all correspondence relative to said application to the following address:

University of California
Office of Technology Transfer
1111 Franklin Street, 5th Floor
Oakland, California 94607-5200

Dated: September 29, 2000

ASSIGNEE: THE REGENTS OF THE UNIVERSITY OF CALIFORNIA

Signature: _____

Typed Name: Tim W. Wan

Title: Licensing Officer
Office of Technology Transfer
The Regents of the University of California
1111 Franklin Street, 12th Floor
Oakland, California 94607-5200